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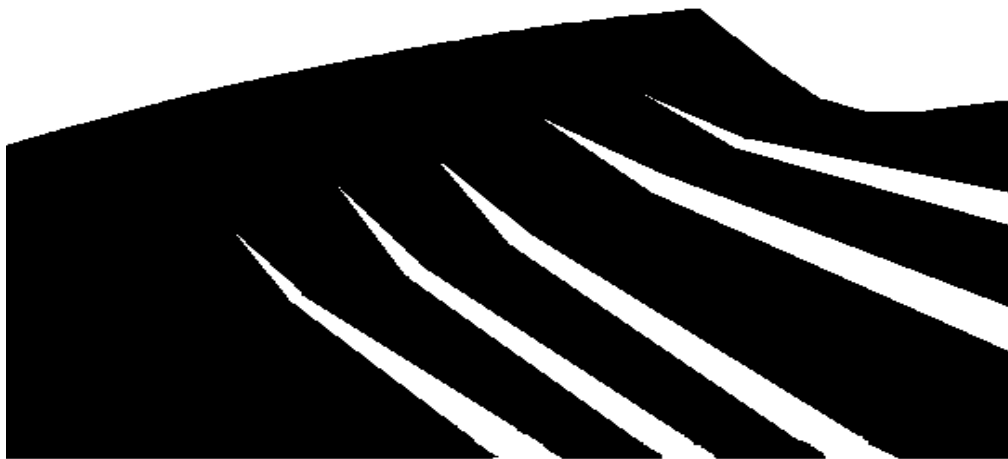
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LANL-EES-DP-116, R2

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QUANTITATIVE X-RAY DIFFRACTION DATA REDUCTION PROCEDURE

LOS ALAMOS QUALITY PROGRAM



APPROVAL FOR RELEASE

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Los Alamos

Yucca Mountain Site
Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0-R1	1/22/89	N/A	Not applicable, history of revision page not in use.
R2	01/21/97	All	Revised to comply with LANL-YMP-QP-06.3 requirements. Revision 1 of this procedure was previously identified as TWS-ESS-DP-116.

Los Alamos

Yucca Mountain Site

Characterization Project

QUANTITATIVE X-RAY DIFFRACTION DATA REDUCTION PROCEDURE

1.0 PROCEDURE

The purpose of this procedure is to describe the methods, procedures, and documentation used when performing Quantitative X-Ray Diffraction (QXRD) analyses.

2.0 SCOPE

This procedure applies to QXRD analyses conducted for the Yucca Mountain Project.

3.0 REFERENCES

- LANL-YMP-QP-02.7, Personnel Training
- LANL-YMP-QP-03.5, Documenting Scientific Investigations
- LANL-YMP-QP-03.20, Software Configuration Management
- LANL-YMP-QP-17.6, Records Management
- LANL-EES-DP-56: Brinkmann Automated Grinder Procedure.
- LANL-EES-DP-16: Siemens X-Ray Diffraction Procedure.
- LANL-EES-DP-24: Procedure: Calibration and Alignment of the Siemens Diffractometers
- LANL-EES-DP-101: Sample/Speciman Collection, Identification and Control for Mineralogy-Petrology Studies.
- Powder Diffraction File, Search Manual, Fink Method, Inorganic, Publication SMF-26 (Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania, 1976) 1017 pp.
- Mineral Powder Diffraction File, Data Book (Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania, 1986) 1396 pp.
- QUANT – Quantitative X-Ray Diffraction Analysis Program, Los Alamos National Laboratory, Users Manual.
- Chung, F.H., (1974): "Quantitative Interpretation of X-Ray Diffraction Patterns of Mixtures. I. Matrix-Flushing Method for Quantitative Multicomponent Analysis," *Journal of Applied Crystallography* 7, 519-525.
- Chung, F.H., (1974): "Quantitative Interpretation of X-Ray Diffraction Patterns of Mixtures. II. Adiabatic Principle of X-Ray Diffraction Analysis of Mixtures," *Journal of Applied Crystallography* 7, 526-531.

4.0 DEFINITIONS

QXRD – Quantitative X-Ray Diffraction

Machine Custodian – The person who is in charge of an instrument and responsible for the maintenance and safety of the instrument.

Software Technical Contact – The person whose name appears as the technical contact on the Software Summary Form for the software or software package.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- The Principal Investigator (PI)
- Users of this Procedure

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, Section 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principles

Quantitative mineral abundances can be obtained from x-ray powder diffraction data using the procedure published by Chung (1974). Our in-house computer program, QUANT, incorporates Chung’s procedure and produces acceptable results when the program is operated and peak intensities are obtained as outlined in the QUANT instruction manual.

6.2 Equipment Hardware/Software

- Brinkmann Grinder, operated in accordance with DP-56.
- Siemens Diffractometers, operated in accordance with DP-16.
- DIFFRAC500 - Siemens Commercial X-Ray (Data) Package.
- QUANT - LANL QXRD analysis program.
- GRAPHINT - LANL Graphical Peak Integrator.

6.2.1 Equipment Malfunctions

N/A

6.2.2 Safety Considerations

N/A

6.2.3 Special Handling

All equipment storage, shipping, and handling considerations are covered in the DPs appropriate to the piece of equipment.

It is important that analysts follow the appropriate procedures for sample preparation and X-raying the sample, and that they follow the instructions in the QUANT user's manual. In addition, if an internal standard is used, weighing and mixing must be conducted with care to ensure no loss of sample or standard that would affect the sample:standard ratio.

6.3 Preparatory Verification

The phases in the sample are identified by comparing the peaks of the sample run to matched peaks in search files (Powder Diffraction File, Search Manual), to JCPDS files (Mineral Powder Diffraction File, Data Book), or to calculated or pure mineral standard patterns.

6.3.1 Hold Points

6.3.2 Calibration

Calibration of the Siemens X-Ray Diffractometer shall be conducted in accordance with DP-24. The computer program QUANT will be verified using known mixtures of mineral standards in accordance with the QUANT instruction manual. Intensity calibration is not necessary since 1.0 μ m corundum powder is normally added as an internal standard in a known ratio or intensity ratios are used with the analyses constrained to total 100%.

6.3.3 Environmental Conditions

6.4 Control of Samples

Samples will be tracked, handled shipped, and stored in accordance with DP-101.

6.5 Implementing Procedures

6.5.1 Sample Crushing and/or Shatterboxing

These operations, if necessary, shall be conducted and documented in accordance with the applicable detailed procedures.

6.5.2 Sample Grinding

This operation shall be conducted in accordance with DP-56.

6.5.3 Mixing Sample with an Internal Standard

1.0 μm alumina powder shall be mixed with a sample by weight if an internal standard is desired. The exact ratio of the mixture is not important as long as the ratio is known and recorded (we normally use 80:20 Sample:Standard). This operation shall be documented in a controlled YMP logbook and shall include the following information: Date, full sample label, grams of sample, grams of alumina powder, and the signature of the person conducting this operation. Vessels containing a sample with an internal standard shall be labeled as such.

6.5.4 X-raying the Sample

The sample mount is prepared, X-rayed, and the data obtained in accordance with DP-16.

6.6 Acquisition and Reduction

6.6.1 All QXRD calculations shall be conducted using the most current version of the computer program QUANT in accordance to the most current corresponding instruction manual for QUANT. QUANT and the records generated by QUANT shall be maintained under software configuration management in accordance with QP-03.20.

6.6.2 Output from QUANT shall include the date of analysis, sample label, analyst's name, the quantitative method being used (internal or external standard method), the version of QUANT being used, and the actual analysis: (the mineral phases identified, their weight percent, and error bars for the weight percent).

6.7 Potential Sources of Error and Uncertainty

Potential sources of error are outlined in the QUANT instruction manual. Potential sources of error include but are not limited to: an amorphous substance in the sample, the presence of an unidentified phase, varying

chemistry of a solid solution phase resulting in variations in the reference intensity ratios, preferred orientation for a phase, peak overlaps, background determination for a peak.

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

- 8.1 Accept/Reject criteria for an individual analysis are outlined in the QUANT instruction manual. Typically, an analysis is rejected if the total significantly deviates (~5-10%) from 100%, if 2 peaks are entered for a mineral phase and the weight percents calculated for each peak are significantly different, or the mineral percentages calculated are in poor agreement with some other empirical method (i.e., CIPW normalization). It is up to the analyst to determine whether the analysis is acceptable for the work being conducted.
- 8.2 The logbook entries for sample preparation and Siemens diffractometer runs and the quantitative analysis data file stored permanently on magnetic media shall constitute evidence that the procedure has been adequately followed and satisfactorily accomplished.

9.0 TRAINING

- 9.1 Prior to conducting work described in Section 6.0, the user requires training to this procedure.
- 9.2 Training to this procedure is accomplished by “read only.” Training will be documented per QP-02.7.

10.0 ATTACHMENTS

N/A